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PLANSEE - SEMINAR

'De re metallica'

ABSTRACTS OF
PAPERS

Reutte/Tyrol
June 22 - 26, 1952

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Section:
"Physical Metallurgy"

Dr.M.AUWÄRTER, Balzers, Liechtenstein

Stability of Inorganic Compounds in High Vacuum

The processes taking place during the melting of inorganic compounds, particularly of oxides in high vacuum, are demonstrated for a number of substances. The dissociation pressure of oxygen, as well as possible reactions with the environment, play a decisive part. The results lead to conclusion regarding the mechanism of melting and sintering of metals, alloys and metallic compounds in high vacuum.

Prof.Dr.H.FORESTIER, University Strasbourg, France

Effect of Adsorbed Gases on the Reactivity of Solid Surfaces

The effect of gas adsorption on the stability of crystal lattices was investigated. This effect is evidenced by:

1) Change of reactivity of solid oxides
2) Change of transformation temperature of polymorphous substances in powder form;
3) Change of melting point of crystalline substances.

The experimental data permit a theoretical interpretation of these phenomena. The gas, which is adsorbed by Van der Waals forces, effects changes in the stability of the energy levels of the solid surface.

Dr.A.KOFLER, Innsbruck, Austria

Micro-Thermal Analysis

I. Contact Method: A microfilm demonstrates the behavior of binary system upon formation of (a) a simple eutectic; (b) one or more molecular compounds; (c) miscibility gaps of liquid phases; and (d) solid solutions.

II. Fusion and Crystallization of Solid Solutions: A second microfilm shows the homogenization of solid solutions upon heating and cooling within the interval between solidus and liquidus. It is demonstrated that this homogenization is not a pure diffusion process since periodical transformations play a contributing part. A homogenization of zone crystals within a few hours is possible only in the presence of small amounts of melt.

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Prof.Dr.R.MITSCHE, Montanistische Hochschule Leoben, Austria

Uniform Hardness Test Using Sintered Hard Metal Penetrators

Technical hardness test methods are generally based on the determination of the "indentation hardness". In these methods, three types of penetrators are employed; either balls (Brinell), cones (Ludwik,Rockwell), or pyramids (Vickers), and accordingly a series of different hardness scales is used. There are certain conversion possibilities, but a uniform hardness test would represent marked progress.

As the result of extensive experimentation, covering the range from soft pure aluminium to fully hardened steels, it is shown that a 136°-pyramid of sintered hard metal permits the development of a uniform indentation test (pre-loading procedure). The evaluation can be based either preferably as on depth of penetration or on the computation of the Vickers hardness number in $kg./mm.^2$

Although a number of considerations favor the use of a set of different loads,it is suggested to employ one load only, preferably 150 (140 + 10) kg.

The use of a 120°-cone, instead of the 136°-pyramid, is possible but in general less advantageons.

Prof.Dr.Ing.H.NOWOTNY, University Vienna, Austria

Bonding in Intermetallic Phases

The present view and theories of bonding in lattices of intermetallic phases are reviewed. Based on a discussion of series of isomorphous compounds, it is attempted to distinguish between the different types of bonding. In particular, it is investigated to what extent simple relationships can be expected between the type of bonding and specific properties such as hardness.

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Prof.Dr.G.-M.SCHWAB, University Munich, Germany

Electron Theory of Mechanical Strength

An approximate theoretical treatment of the temperature coefficient of the penetration hardness of metals permits the derivation of the energy factors determining plastic deformation. While previously the strength could be related to atomic dimensions only, the new theory leads to a relation between the formation of dislocation and the electron structure of metals.

Prof.Dr.W.SEITH, University Münster, Germany

New Concepts of the Diffusion in Metals

New experimental results in the field of diffusion in metals are reviewed, and the modern concept of the interchange mechanism, as derived from these results, is discussed. Special attention is given to the conditions prevailing during the sintering of metal powders.

Prof.Dr.F.SKAUPY, Technical University Berlin, Germany

Present Views on the Nature of Metals and Their Significance for Metal Ceramics (Powder Metallurgy)

The present views on the nature of metals are reviewed, special attention being given to interatomic distances and the properties of thin layers. The concepts are applied to some problems of powder metallurgy.

Prof.Dr.A.G.SMEKAL, University Graz, Austria

Ideal and Real Crystals

Structure-insensitive and structure-sensitive crystal properties. Ideal crystal and the problem of the stability of extended crystal lattices. The microstructure of real crystals, lattice deficiencies and foreign matter. Effect of lattice structures and chemical bonding types (e.g., on selfdiffusion and plastic deformation). Characteristics of ideal structure elements.

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Prof.Dr.R.WALZEL, Montanistische Hochschule Leoben, Austria

Principles of Dry Metallic Wear

The wear process is considered as being due to friction forces in excess of the tensile and shear strength of the surface layer. It is difficult to predict wear resistance values and to draw conclusions from laboratory tests to the performance to be expected in service. These difficulties are due to the complicated dependence of friction forces on many factors as well as to the fact that the tensile and shear strength of the surface layer is not constant but is determined by the changes taking place during wear. It is attempted to correlate the various factors involved. The special conditions determining the wear of sintered materials are pointed out.

Section:

"General Powder Metallurgy"

Dr.H.S.CANNON and Prof.Dr.F.V.LENEL, Rensselaer Polytechnic Institute, Troy, N.Y., U.S.A.

Some Observations on the Mechanism of Liquid Phase Sintering

In this paper several experiments on liquid phase sintering are reported and some hypotheses concerning the mechanism of liquid phase sintering are offered for discussion. They are based on our own experimental observations and those of previous investigators. The rate of densification and the microstructure of sintered compacts of the following powder mixtures were determined:

Iron-Copper
Copper-Bismuth
Copper-Lead
Tungsten-Copper

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The system tungsten-copper differs from the others investigated in that the solid phase has only negligible solubility in the liquid phase during sintering. Nevertheless, considerable densification is observed, the extent of which depends upon the sintering conditions. Predictions based an considerations of surface and interfacial tensions are made on how complete densification may be expected in such a system. The mechanism of densification in the two types of systems are compared those with and those without appreciable solubility of the solid phase in the liquid phase.

In the systems iron-copper, copper-bismuth and copper-lead it is observed that under certain conditions the densification of the compacts is incomplete even though they are sintered for long times. The cessation of densification is attributed to the formation of a rigid skeleton of the solid phase during sintering. It is postulated that rigid skeletons can be formed when the ratio of the solid-solid to the solid-liquid interfacial tensions is favorable. Experiments are described supporting this rigid skeleton theory. The difference between the rigid skeletons in the systems studied in this investigation and those observed in certain cemented carbide compositions is discussed.

Prof.G.J.COMSTOCK, Stevens Institute of Technology, Hoboken,N.Y., U.S.A. and Dr.F.H.CLARK, President's Materials Policy Commission

The Development of High Strength Heat Treatable Products from Alloy Powders

Continued progress in the United States toward the development, production and application of fully alloyed metal powders as one of the most significant factors in widening the field of powder metallurgy. The types of materials now being produced; their general characteristics, the present preferred methods of fabricating pre-alloyed powders, and the potentialities presented by the development of high-strength, high-density, heat-treatable materials. Progress in the general production of metal powder parts and charts indicating the growth of the industry. Examples of new products.

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Prof. Dr. E. CREMER, University Innsbruck, Austria

Determination of the Adhesive Force (Haftkraft) of Metal Powders

The measurement of the glide angle of a powder dusted on a solid support permits the determination of a characteristic value which is termed "adhesive force" (Haftkraft). This value depends on the surface properties of both powder and support, and is inversely proportional to the particle diameter of the powder.

Dr. H. H. HAUSNER, New York, U.S.A.

The Effect of Lattice Changes on the Sintering Process

Since the rate of sintering depends among others on the mobility of the atoms within the lattice, reactions which create lattice imperfections, lattice disturbances or complete lattice changes may further affect the rate of sintering. The following reactions are discussed in the paper, and their effect on the sintering process are analyzed:

1. Reductions Example: $MO + H_2 \longrightarrow M + H_2O$
2. Decompositions Example: $MH \longrightarrow M + H_2$
3. Phase Changes Example: $M\alpha \longrightarrow M\beta$

Experimental work described in the paper:

a. The effect of heating on nickel powder compacts in a stream of hydrogen at temperatures up to 100°C and the changes in electrical resistivity.
b. The heating of zirconium hydride powder compacts in vacuum, the decomposition of the hydride into zirconium and its effect on densification and grain size.
c. The thermocycling of an iron powder compact around the phase transformation temperature and its influence on density, shrinkage and grain size.

The experimental results show that the above described three types of lattice changes effect the sintering procedure considerably although the changes do not always occur in a similar way.

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Prof.Dr.G.JANTSCH and Dr.F.ZEMEK, Technical University Graz, Austria

Deposition of Chemically Pure Vanadium from the Gas Phase

The observation of black metallic mirrors in experiments on the disproportionation of tri- and dihalogenides of vanadium initiated attempts to produce pure vanadium powder. Vanadium tetrachloride was prepared by chlorination of ferrovanadium, was fractionated and distilled. Mixtures of the purified tetrachloride with purest hydrogen were introduced into a furnace preheated to 700°C. Purest hydrogen is introduced from the opposite direction and accelerates reduction by eddy formation.

Chemical and spectrochemical analysis indicated that the vanadium powder thus prepared contains 99 % V, remainder hydrogen. Debye-Scherrer diagrams show a cubic body-centered lattice, a = 3,0204 Å. Measurements in the electron microscope (15,000 x) give the particle diameter as 0,2-0.3 micron. Electron-diffraction results agree with the x-ray data.

Dr.W.J.KROLL, Corvallis, Oregon, U.S.A.

The Production of Metal Powders by Fusion Electrolysis

The production of metal powders by fusion electrolysis is a much more universal method than the fused bath electrolysis with molten cathode deposit. The reasons why the first method has been used only in special cases up to now, are discussed. The difficulties involved concern mainly the cell construction, which might require the use of a baffle, the problem of a suitable electrolyte, the one of the anode which might either be insoluble or soluble, and the question of continuous and semicontinuous operation and its reflection on cell design. The difficulties in producing titanium powder by fusion electrolysis are stressed and examples are given for obtaining tungsten, molybdenum, manganese, iron and alloy powders by this method, using insoluble, and in some cases, soluble anodes.

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Dr.G.RITZAU, Krefeld, Germany

Contribution to the Physical Analysis of the Sintering Process

Methods which permit the elucidation of the sintering mechanism by the direct measurement of changes taking place during sintering deserve special attention. The electrical conductivity and the thermoelectric potential appear particularly suited for a "physical analysis" of this type. Whereas the variation of the electrical conductivity is determined by both the change in porosity and changes of the microstructure which may be effected by diffusion processes, the variation of the thermoelectric potential is independent of porosity. The principles of the method are discussed for binary systems which exhibit either complete mutual solubility (copper-nickel) or complete mutual insolubility (copper-iron) in the solid as well as in the liquid states. The poor sintering performance of nickel is, among others, clearly indicated by this analysis. For sintered hard metals and sintered magnets, it is shown that the method is capable of recording the course of sintering in complex alloys.

Prof.Dr.F.SAUERWALD, University Halle/Saale, Germany

Surface Layers, Particularly Oxide Layers, and their Effect on Sintering (Experiments by M.Clasing)

In studying the effects of surface layers of different thickness on compacting, sintering and the mechanical properties of sintered materials, the stability of the surface layers must be taken into consideration. It has, e.g., been established that oxide layers formed by oxidation of copper in air are not stable under conditions encountered during sintering. The first part of the paper is concerned with the establishment of the factors responsible for the stability of oxide films on copper.

The experimental results show that in the case of normal copper which is not saturated with oxygen the diffusion from the surface into the interior at high temperatures is mainly responsible for the instability of oxide films.

The second part of the investigation deals with the effect of stable and unstable oxide films on the mechanical strength of sintered copper compacts. It was found that surface oxides on

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powder particles, and particularly unstable surface oxide films, may, under certain conditions, have a favorable effect on the strength of sintered compacts. In other instances, the well-known unfavorable effect of oxide layers was confirmed.

Prof. Dr. A. von ZEERLEDER, Neuhausen, Switzerland

New Developments in the Powder Metallurgy of Aluminum

The rate of sintering of aluminum is slow and this metal therefore, represents a special case in the field of powder metallurgy and has so far been rarely used in the form of sintered products. The apparent density of commercial aluminum powder ranges between 0.1 and 0.3 so that an excessively large decrease in volume occurs during processing by powder metallurgy. The powder used for the manufacture of sintered aluminum - trade name SAP (Sintered Aluminum Powder) - is prepared by a special process and has, in spite of very fine particle size, an apparent density between .8 and 1. This powder is first cold prepressed at a pressure between 14 and 35 tsi. After being sintered at 500 to 600°C., the compact is hot pressed at 35 tsi. Thus prepared cylinders are extruded at 500 to 600°C. at a pressure of 35 to 70 tsi. The extruded material can then be either die-forged or hot-swaged. Instead of being extruded, the cylinders may also be directly die-forged. The outstanding property of the SAP products is the very high strengths and particularly the hot strength. The strength is dependend on the grain size and reaches, for powders of a particle size smaller than one micron, values exceeding 50,000 psi. Since each individual particle is coated by an oxide film, no grain growth is observed even after long-time heat treatment and the creep strength of SAP at 400°C. is by far superior to that of all aluminum alloys. However, in the manufacture of SAP, the oxide content must be closely controlled since higher oxide content increases the strength but, at the same time, reduces elongation and toughness. Due to the very fine grained structure, SAP also exhibits a superior fatigue strength at high temperatures.

The outlined characteristics indicate the suitability of SAP for applications in the temperature range between 200 and 400°C.

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Section:

"Hard Metals"

Ing. C. BALLHAUSEN, Krefeld, Germany

Properties of Hard Metal Alloys in Comparative Presentation

The properties of hard metal alloys are plotted as space co-ordinates on an rectangular base having the volume ratio WC:TiC as abscissa and the Co content in area-percent as ordinate.

Data on compressive strength, modulus of elasticity, hardness, modulus of rupture, coefficient of thermal expansion and cutting characteristics are plotted for WC-TiC-Co-base hard metals.

A suggested standardization provides specifications according to WC-TiC ratios and Co contents.

Doz. Dr. E. FITZER, Technical University Vienna, Austria

High-Temperature Materials by Silicizing Tungsten and Molybdenum

The high-melting metals tungsten and molybdenum, which exhibit high-temperature strength, can be protected against oxidation by alloying with silicon; tungsten can be protected up to 2000° and molybdenum up to 1800°C. The silicides of these metals have practical importance either in the form of thin surface coating layers or in the form of bulk materials produced by techniques of powder metallurgy. Alloy formation restricted to the surface can be effected by catalytic reduction of silicon tetrachloride with hydrogen. This heterogeneous reaction can also be utilized for the manufacture of $MoSi_2$ powders. The alloying of the pure components molybdenum and silicon in the solid state proceeds too slowly for the technical production of surface layers, but can be utilized for the preparation of sintered alloys. The alloys of this group appear promising not only as heating elements but also for gas turbine and rocket applications.

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Prof. V. D. FRECHETTE, Alfred University, Alfred N.Y., U.S.A.

Ceramics, Metals, and Cermets

Experience with ceramic-coated metals (vitreous enamels), metal-coated ceramics (mirrors, glass-to-metal seals), and metal-reinforced ceramics (Metalkase brick, wire glass) throws light on the newer field of metal-ceramic mixtures and suggests considerations to be applied in designing cermet bodies and coatings for high-temperature applications.

Conversely, recent developments in cermets are suggestive of fresh attacks to old problems in the ceramic field.

Prof. Dr. G. F. HÜTTIG, Technical University Graz, Austria

Problems of the Preparation of Purest Titanium Carbide

In preparing pure titanium carbide, as well as other metal carbides, in general the following requirements should be met:

(1) the chemical composition should correspond to the chemical formula;
(2) the free-carbon content should be as low as possible;
(3) the particle size distribution should be within certain limits.

The modern principles of inorganic chemistry, which are mainly based on the activated-state concept, are reviewed. According to the choice of the gas atmosphere, in which the carbide formation takes place, this atmosphere may have a purely catalytic effect, may act as pace-maker for the main reaction, or may participate in the reaction. In the last case, it must be distinguished between a direct carburization by the gaseous agent and an indirect effect which can be described as the creation of equilibrium conditions which are prohibitive for the existence of free graphite.

Another series of principles is based on the use of chemically active and therefore reactive starting materials, or the use of starting materials which during the course of the reaction produce - in statu nascendi - the components required for the carbide formation.

The experimental material, part of which has not been published previously, was obtained at the Institute for Inorganic and Physical Chemistry of the Technical University Graz, mainly by D. Schuler, V. Fattinger and K. Kohla.

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Dr. R.KIEFFER, Reutte/Tyrol, Austria

Formation of Solid Solutions of Hard Metals

The term "Hard Metals" refers to the carbides, nitrides, borides and silicides of the transition metals of the fourth to sixth groupe of the periodic table. The main constituents of modern tool materials are the carbides of the transition metals and particularly binary and ternary solid solutions of these carbides. The complete solid solubility of the isomorphous carbides of the metals of the fourth and fifth groups, as well as the partial solubility between these carbides and those of the sixth-group metals, is discussed. The cubic nitrides of the transition metals of the fourth and fifth groups form, with few exceptions, solid solutions with each other and with the isomorphous carbides of the same groups. Borides and silicides of the transition metals have recently found application in high-temperature materials. While the carbides and nitrides have in general simple strucutres, the borides and silicides form a large number of hard metal phases with comparatively complicated structures. Isomorphous borides as well as silicides tend towards formation of solid solutions. It is attempted to follow, metallographically as well as by microhardness measurements, the diffusion processes involved in the formation of solid solutions.

Dr. R.KIESSLING, Söderfors, Sweden.

Investigations on Ternary Systems Me_1-Me_2-B, and a Discussion of the Relative Strength of the Bond Transition Metal-Boron

A summary of the work at Uppsala on ternary systems, containing two transition metals and boron, will be given.

For the systems Mn-Fe-B, Mn-Co-B, Mn-Ni-B, and Fe-Co-B the range between 33 and 50 atomic % boron has been studied. An equilibrium between two phases $(Me_1Me_2)_2B$ and $(Me_1Me_2)B$ exists and the directions of the tie-lines show, that the metal with the lowest atomic number always is concentrated in the phase, richest in boron. Distribution ratios for the metals Mn, Fe, Co,

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amd Ni between the boronrich $(Me_1Me_2)B$ and the boronpoor $(Me_1Me_2)_2B$ phase have been calculated and a graphic representation of the equilibrium data will be presented. These data show, that the strength of the metal-boron bond within a certain transition serie decreases with increasing atomic number of the metal.

The structures of ternary alloys of the type $(Me_1Me_2)B_2$ are also beeing studied at the present time and some preliminary results from these investigations will be reported.

Prof.Dr.J.T.NORTON, Massachusetts Institute of Technology Cambridge, Conn, U.S.A.

The Ternary System Tungsten-Carbon-Cobalt

Although alloys of tungsten monocarbide and cobalt have been extensively studied and widely used in practice, there is still considerable uncertainty about the nature of the true ternary phase diagram. Experimental investigations have established the ternary isothermal section at 1400°C and have shown in addition to the well known eta phase, two new ternary phases corresponding approximately to the formulae $Co_3W_6C_2$ and $Co_3W_{10}C_4$. The former has a cubic lattice and the latter hexagonal. Isomorphous phases were observed in the analogous systems containing iron or nickel. The reactions leading to the formation of these phases have been determined and a tentative phase diagram proposed. Evidence for the existence of stable and unstable equilibria is presented. On the basis of the proposed diagram, the reactions during the sintering of alloys of tungsten carbide and cobalt are examined and some of the details of the structure and properties of cemented carbide compositions are discussed.

Dr.T.RAINE and Dr.R.EDWARDS, Manchester, England

The Solid Solubilities at 1250°C of Various Carbides in the Bonding Metals Cobalt, Nickel and Iron

The solid solubilities at 1250°C of the Carbides of W, Ti, Ta, Nb, Mo, V and Co and also TiC-WC mixed crystals in Co, Ni and Fe respectively have been determined by metallographic and X-ray methods.

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Doz.Dr.E.M.ONITSCH-MODL, Montanistische Hochschule Leoben,Österreich

The Micro-Hardness Test Employed as an Auxiliary on the Examination of Sinter Changes in Complex Systems

The micro-hardness test can serve as a simple and easily mastered method to examine alloying processes, which incorporate hardness changes. In the case to be discussed alloying process incurred during the sintering of Fe-C alloys, which contain the carbide forming elements Cr, W, Mo and V, will be observed by means of the micro-hardness test.

Diffusion processes, which take place during the sintering dependant on the sintering temperature, both in the basic material and during the carbide formation, could be registered easily by micro-hardness tests. Hereby it was found that the micro-hardness test could both be applied advantageously as such and as a supplement for metallographical methods and for the examination of very fine structures.

Applications and limits of the micro-hardness in respect of recording of sintering processes, will be shown on some examples.

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